



Europäisches Patentamt  
European Patent Office  
Office européen des brevets



Publication number:

**0 538 861 A1**

## EUROPEAN PATENT APPLICATION

Application number: **92118098.0**

Int. Cl.<sup>5</sup>: **H01J 37/20**

Date of filing: **22.10.92**

Priority: **24.10.91 JP 277450/91**  
**18.06.92 JP 159133/92**

Date of publication of application:  
**28.04.93 Bulletin 93/17**

Designated Contracting States:  
**DE GB NL**

Applicant: **HITACHI, LTD.**  
**6, Kanda Surugadai 4-chome**  
**Chiyoda-ku, Tokyo 101(JP)**

Inventor: **Aoyama, Takashi**  
**55-1, Minamidai, Tokaimura**  
**Naka-gun, Ibaraki-ken(JP)**  
Inventor: **Hosoi, Kishu**  
**3-14, Higashinamekawacho-3-chome**  
**Hitachi-shi(JP)**

Inventor: **Misawa, Yutaka**

**1229-1, Tabiko**  
**Katsuta-shi(JP)**

Inventor: **Kimoto, Koji**  
**Mikanohararyo A109, 20-1,**  
**Mizukicho-2-chome**  
**Hitachi-shi(JP)**

Inventor: **Isakozawa, Shigeto**

**1319-1, Tabiko**  
**Katsuta-shi(JP)**

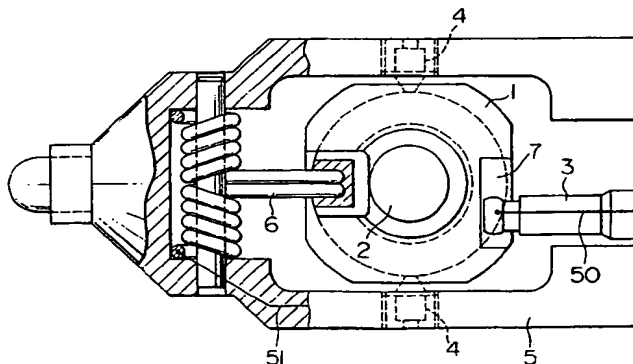
Inventor: **Ueda, Kazuhiro**  
**Yuhoryo, 20-3, Ayukawacho-6-chome**  
**Hitachi-shi(JP)**

Representative: **Strehl, Schübel-Hopf,**  
**Groening**  
**Maximilianstrasse 54 Postfach 22 14 55**  
**W-8000 München 22 (DE)**

**Electron microscope specimen holder.**

In order to control the position of a specimen on a specimen holder with high accuracy, a specimen cartridge (1) is made of a good thermal conductor, an outer frame (5) is made of heat insulating material and a connecting rod is made of a poor thermal conductor. The temperature distribution of the specimen (2) becomes uniform and the temperature drift is reduced. Further, thermal expansion of a specimen cartridge tilting rod (3) does not affect the tilting angle. These features make it possible to perform various highly accurate observations and measurements with an electron microscope.

**FIG. 2**



## BACKGROUND OF THE INVENTION

### Field of the Invention

5 The present invention relates to an electron microscope specimen holder and, more particularly, to an electron microscope specimen holder which enables the specimen position to be stable even if heated and cooled.

### Description of the Related Art

10

In case of observing a specimen and performing various measurements (e.g., elemental analysis using characteristic X-rays) with an electron microscope, in order to prevent contamination of the specimen or induce the phase change, it is general to observe and measure the specimen not at room temperature but in the heated or cooled state.

15 As the temperature is elevated or lowered, the specimen position is changed due to thermal expansion or contraction of the holder, and accurate observation and measurement will be difficult. To cope with this, there have conventionally been known the following specimen or sample holders.

In a first type of specimen holder, the holder is made of plural materials whose thermal expansion coefficients are different from each other and they expand (shrink) in opposite directions so that a net shift 20 of specimen (sample) position caused by thermal expansion is cancelled. In such a case, accurate observation or measurement can be performed (U.S. Pat. No. 3,896,314, U.S. Pat. No. 4,703,181).

In a second type, the temperature change of the specimen holder is detected by a sensor and a specimen support is moved in an amount corresponding to the thermal expansion so as to keep the specimen position unchanged, thereby enabling accurate observation or measurement (Japanese Patent 25 Unexamined Publication No. 1-197952(A)).

The first type convention is based on an assumption that a uniform temperature distribution can be obtained over a wide area. In real sample (specimen) holders, however, thermal conduction of various materials is important and there exist temperature profiles which change in various forms, resulting in an error of the specimen position.

30 In the second type convention, the actual position change of the specimen is not detected, and it is not easy to keep the specimen position unchanged.

Further, in any case, no consideration has been given to the accurate rotation about a tilt axis or axes of the specimen, particularly the tilt axis perpendicular to the axis of the specimen holder.

As described above, the prior art involve the problem that it is difficult to keep the specimen position 35 unchanged, in the heating specimen holder accompanied with parallel movement and rotary driving.

## SUMMARY OF THE INVENTION

### Object of the Invention

40

An object of the present invention is to make it possible to control the specimen position with high accuracy particularly in a heating specimen holder thereby enabling to actualize highly accurate observation and measurement with an electron microscope.

### Statements of the Invention

An electron microscope specimen holder according to an aspect of the present invention has a specimen support such as a specimen cartridge within an outer frame and the specimen support has larger thermal conductivity than that of material of the outer frame.

50 Particularly, it is preferred that the coefficient of thermal expansion of the material for the outer frame is in the range of  $5.0 \times 10^{-7} \sim 1.2 \times 10^{-5}/K$ . Further, it is preferred that the specimen support is made of titanium or stainless steel and the outer frame is made of sialon, cordierite or titanium.

Further, an electron microscope specimen holder according to a further aspect of the present invention comprises an outer frame, a specimen support such as a specimen cartridge within the outer frame and 55 tilting pivots serving to incline or tilt the specimen support, wherein a thermal conductivity of material of the specimen stage is greater than that of material of the outer frame and a coefficient of thermal expansion of material of the tilting pivots is in the range of  $5.0 \times 10^{-7} \sim 1.2 \times 10^{-5}/K$ .

Moreover, an electron microscope specimen holder according to a further aspect of the present invention is of the type that a specimen set on a specimen support such as a specimen cartridge is moved in a plane and driven rotatively and a rod serving to control an angle of the specimen support is in contact with the specimen support on a torsion or twistedly warped surface.

In addition, an electron microscope specimen holder according to a further aspect of the present invention comprises a specimen support such as a specimen cartridge, a rod serving to control an angle of the specimen support and a pressure spring, wherein the specimen support has a double-layer structure including a platform on which a specimen is to be placed and a heater, and the rod and the pressure spring serve as power supply lines for the heater as well. In this way, by omitting specific power supply lines, it is possible to control the tilt angle with high accuracy.

Particularly, it is preferred that the specimen support is of a stacked structure including a metal portion and a ceramic heater.

Further, an electron microscope specimen holder according to a further aspect of the present invention has a coupling or joint structure including a high temperature part and a low temperature part, wherein the high temperature part is made of heat insulating material and the low temperature part is made of low thermal expansion material.

Moreover, an electron microscope specimen holder according to a further aspect of the present invention comprises a specimen support such as a specimen cartridge, wherein a thermal conductivity of material of an inner part of the specimen support is greater than that of material of an outer part thereof.

In addition, an electron microscope specimen holder according to a further aspect of the present invention is of the type that a specimen support such as a specimen cartridge is disposed within an outer frame and is heated in use, wherein a temperature gradient between the specimen support and the outer frame is steep when the specimen holder is in use.

Furthermore, an electron microscope specimen holder according to a further aspect of the present invention is of a type that a position of a specimen is changed, wherein an amount of change of the specimen position is in the range of 0.02 ~ 0.10 mm/sec.

Besides, according to another aspect of the present invention, there is provided an electron microscope in which one of the above-mentioned electron microscope specimen holders is mounted.

Further, an electron microscope specimen holder according to a further aspect of the present invention is adapted for use in observing a state of a specimen placed on a specimen support such as a specimen cartridge by applying an electron beam to the specimen and comprises a heater at a bottom of the specimen support for heating the specimen on the specimen support up to a predetermined temperature, a rod for controlling an angle of the specimen support and a pressure spring for supporting the specimen support, wherein the rod has a shape that makes a contact surface between the rod and the specimen support stationary in the vertical direction and electric power is supplied to the heater through the rod and the pressure spring.

In addition, an electron microscope according to another aspect of the present invention is of a transmission type which comprises an electron gun for producing an electron beam, an optical system for applying the electron beam to a predetermined range at a fixed amount, a specimen or sample holder on which a specimen is to be placed so as to be observed as being applied with the electron beam, another optical system for observing a state of the specimen on the specimen holder and an operation control system, wherein the specimen holder has a structure that a specimen support such as a specimen cartridge is disposed within an outer frame, a thermal conductivity of material of the specimen support is made greater than that of material of the frame, and the specimen support is provided at a lower part thereof with a heater to which electric power is supplied through a pressure spring serving to hold the specimen stage and through a rod serving to adjust a tilting or inclination of the specimen holder.

Further, an electron microscope heating specimen holder according to a further aspect of the present invention comprises a specimen holder and a bearing or connecting rod, wherein a thermal conductivity of material of the rod is smaller than that of material of a main body of the specimen holder.

Particularly, it is preferred that the thermal conductivity of the rod is in the range of 15 ~ 20 W/mK.

In addition, an electron microscope heating specimen holder device according to a further aspect of the invention comprises a specimen holder for holding a specimen and a bearing or connecting rod serving to finely control a position of the specimen, wherein the specimen holder is made of phosphor bronze and the rod is made of SUS304.

Further, an electron microscope heating specimen holder according to a further aspect of the invention is of a type that a heater for heating a specimen is disposed at a bottom of a specimen stage support such as a specimen cartridge for supporting the specimen, wherein the heater is a plate heater made by forming a film of high resistance metal on a plate-like insulating member by means of vapor deposition or sintering.

Still further, an electron microscope heating specimen holder according to a further aspect of the present invention comprises a heater formed on an insulating member or members and serving to heat a specimen, wherein the heater is a plate heater made by filling a groove formed in a plate-like insulating member with high resistance metal or by sandwiching high resistance metal between the plate-like insulating members.

In addition, an electron microscope heating specimen holder according to a further aspect of the present invention has a structure that a thermocouple is in contact with a specimen support such as a specimen cartridge, wherein the thermocouple is disposed at a tip end of an elastic lead portion.

Besides, according to another aspect of the present invention, there is provided an electron microscope in which one of the above-mentioned electron microscope specimen holders is mounted.

In the electron microscope specimen holder, it is necessary to control the specimen position with high accuracy. Further, it is also necessary to move or displace the specimen in performing various measurements and observations. The electron microscope specimen holder of the present invention can perform even such movements with high accuracy. Planar movement is made within the X-Y plane while rotary movement is made about x and Y axes with the angle controlled within a range of about  $\pm 15^\circ$ . Namely, the electron microscope specimen holder of the invention is typically a double tilt heating specimen holder in which the specimen can be rotationally displaced about two axes.

The present invention is also directed to a transmission electron microscope which comprises an electron gun of field emission type and is applicable to one in which the electron beam can be converged or focused down to a range of 1 nm in diameter. This type of electron microscope is capable of performing various analyses of a very small range and, particularly according to the present invention, the amount of drift of the specimen position can be reduced to not more than 0.1 nm. The present invention can solve the problem of the prior arts and meet a demand of control with high accuracy.

In other words, typically in the present invention, the specimen support such as a specimen cartridge, of the specimen holder is made of good thermal conductor and the outer frame of the specimen support is made of heat insulator. Further, the specimen support and the specimen support tilting rod are brought into contact with each other on a warped surface. In addition, the specimen support tilting rod is made to serve as the heating power supply line as well.

In the present invention, the specimen holder main body is made of good thermal conductor and the bearing or connecting rod which serves to support the specimen holder from the side of TEM and move the specimen holder slightly is made of such material that has a lower thermal conductivity than the material for the specimen holder. Further, in order to measure the specimen temperature, there is provided the thermocouple in contact with the specimen support under elastic force.

According to the present invention, since the specimen support portion is made of good thermal conductor, the temperature distribution becomes uniform. Further, since the outer frame portion is made of heat insulating material, the temperature rise thereof is suppressed as compared with that of the specimen support portion. In consequence, even if there is caused temperature drift in the specimen support portion, a magnitude of the temperature drift in the outer frame (holder portion) is very small. For this reason, the horizontal position of the specimen portion and the angle of inclination or tilting of the specimen support are affected very little, thereby making it possible to perform accurate observation and measurement with the electron microscope.

Further, by making the specimen support and the specimen support tilting rod come in contact with such outer on a warped surface, the contact surface can be kept in parallel at all times with the direction in which the length of the rod is changed due to thermal expansion. For this reason, even if the length of the rod is changed due to thermal expansion, the angle of inclination or tilting can be kept constant at all times.

Moreover, since power is supplied to the tilting specimen support heater through the rod and the pressure spring, the tilting specimen support (cartridge) and the outer frame can be brought into contact with each other only through the rod, spring and sapphire pivot. For this reason, conduction of heat from the specimen support to the outer frame can be suppressed as small as possible, thereby making it possible to minimize the thermal drift of the outer frame.

In addition, description will be given of the function effected when the specimen holder is made of good thermal conductor and the bearing or connecting rod of a fine adjustment device is made of such material that has a thermal conductivity lower than that of the material for the specimen holder. The specimen holder is supported by the TEM main body by means of the connecting rod and, hence, thermal expansion of the connecting rod directly results in change of the specimen position.

On the other hand, thermal expansion of the specimen holder has substantially no effect on the change of the specimen position since the specimen holder can be moved in the lateral or transverse direction. The time required for all of the specimen holder and the connecting rod to reach the thermal equilibrium

depends on the thermal conductivity of the specimen holder. After reaching the thermal equilibrium, the amount of drift of the specimen position depends on a slight change in the temperature of the connecting rod.

In case that the specimen holder is made of highly thermally conductive material, the specimen holder reaches the thermal equilibrium state in a short time. Therefore, the temperature of the connecting rod reaches the thermal equilibrium state in a short time as well.

In case that the connecting rod is made of poor thermal conductor the temperature gradient becomes steep from the contact portion with the specimen holder so that the temperature of the bearing rod is decreased as a whole. Accordingly, the absolute value of temperature change is reduced, thereby making it possible to reduce the drift of the position.

Next, description will be given of the heating power supplying method and the temperature measuring method. It has been customary to supply power by connecting lead wires directly to the specimen support and measure the thermoelectromotive force of the thermocouple, and however, in the case of the present invention, since there is no possibility that the lead wires stands in the way, it becomes possible to incline or tilt the specimen support with high accuracy. Further, in the specimen holder which has the lead wires, breaking of wire is caused due to fatigue of metal resulting from repeated tilting. In the present invention, however, the lifetime of the specimen holder is extended, thereby solving the conventionally pending problem of short lifetime of the specimen holder.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a sectional view for illustrating the whole construction of a specimen drive portion of an electron microscope to which a specimen holder according to an embodiment of the invention is attached;

Figure 2 is a plan view of a specimen support or cartridge portion of Fig. 1;

Figures 3A and 3B are graphs showing the temperature distribution of the specimen holder;

Figure 4 is a perspective view of the specimen cartridge portion of Fig. 2;

Figure 5 is a sectional view of the specimen cartridge portion of Fig. 2;

Figure 6 is a sectional view of the specimen holder, according to an embodiment of the invention, in which a heater is employed;

Figure 7 is a general construction view of a transmission electron microscope in which the specimen holder of the invention is employed;

Figure 8 is a characteristic view showing the change of the amount of drift with the lapse of time;

Figures 9A and 9B are views for explanation of two kinds of the power supply mechanisms, according to embodiments of the invention, for the heating portion; and

Figure 10 is a view for explanation of the structure for measuring the temperature of the specimen according to an embodiment of the invention.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The foregoing and other objects, features and advantages of the invention will be made clearer from description given below of embodiments of the present invention with reference to the drawings.

Figure 1 is a sectional view for illustrating the whole construction of a specimen drive section of an electron microscope to which a specimen holder or sample holder 8 is attached. As the specimen holder 8 is inserted in a column 13 of the electron microscope, it abuts a fine adjustment device through a connecting rod (bearing rod) 9 and comes to a halt. The fine adjustment device comprises a fine movement shaft 10, a lever or bell crank 11, a screw rod 12 and the like. The system is as shown in Figure 1 in which an electron beam is passed along an electron beam path 14 through an outer frame 5 of a specimen support (cartridge) portion of the specimen holder 8 so as to observe or measure the specimen in respect of its form and the like.

Figure 2 is a plan view showing the details of a specimen support or cartridge (tilting cartridge) of the specimen holder 8.

In Figure 2, reference numeral 1 denotes a specimen cartridge as the specimen support, 2 denotes a specimen, 3 denotes a rod for adjusting inclination or tilting of the specimen cartridge, 4 denote pivots through which the specimen cartridge 1 is supported by an outer frame 5, 6 denotes a pressure spring for keeping the specimen cartridge 1 from moving in the vertical direction, and 7 denotes a contact surface between the specimen cartridge 1 and the rod 3.

It is noted here that the specimen cartridge 1 is made of silicon carbide (SiC). Since the silicon carbide has a large thermal conductivity (62.9 W/mK), the temperature distribution of the specimen becomes uniform.

On the other hand, the frame portion 5 is made of cordierite a thermal conductivity of which is as small as about 1/30 (1.7 W/mK) as compared with the silicon carbide, so that conduction of heat from the specimen carriage 1 to the outer frame 5 is controlled to be low. As a result, in case of heating the specimen 2 up to 200°C, the temperature drift of the specimen 2 is about  $\pm 1^\circ\text{C}$ , and however, since the rise of the temperature of the frame 5 itself is limited to about 110°C the temperature drift thereof is controlled or suppressed to be less than about  $\pm 0.02^\circ\text{C}$ . Therefore, change in the horizontal position caused by thermal expansion is almost negligible.

Next, description will be given of a case that the part of the specimen cartridge of the specimen holder is made of good thermal conductor and the frame 5 of the specimen cartridge 1 is made of heat insulating material.

Figure 3B shows a temperature distribution of a conventional specimen holder obtained when the specimen cartridge 1 and the frame 5 were made of the same material (alumina).

Since the thermal conductivity is not large in the part of the specimen cartridge 1, the temperature distribution of the specimen cartridge is caused to be non-uniform. Alumina has too large a thermal conductivity for the frame 5 so that the whole frame 5 is heated to high temperature, resulting in a temperature drift of a magnitude substantially the same as the specimen portion. This temperature drift affected upon the horizontal position of the specimen portion and the angle of inclination or tilting of the specimen cartridge 1, thereby making it difficult to make precise observation and measurement with the electron microscope.

Figure 3A shows a temperature distribution of the specimen holder according to an embodiment of the present invention.

Since the part of the specimen cartridge 1 is made of good thermal conductor (silicon carbide SiC or titanium), the temperature distribution thereof is uniform. The part of the frame 5 is made of heat insulating material (cordierite or sialon) so that temperature rise thereof is suppressed as compared with the part of the specimen cartridge 1. Therefore, even if a temperature drift takes place in the part of the specimen cartridge 1, the temperature drift of the frame (holder portion) is very small. In consequence, the horizontal position of the specimen portion and the angle of inclination or tilting of the specimen cartridge are affected very little, thereby having made it possible to make precise observation and measurement with the electron microscope. Table 1 shows the heat conductivities of the specimen holders according to the conventional art and examples of the present invention.

Table 1

		Specimen Cartridge	Frame
Conventional Art	Material	Alumina (Al <sub>2</sub> O <sub>3</sub> )	
	Thermal Conductivity (W/mK)	16.8	
Example 1	Material	Silicon Carbide (SiC)	Cordierite
	Thermal Conductivity (W/mK)	62.9	1.7
Example 2	Material	Titanium (Ti)	Sialon
	Thermal Conductivity (W/mK)	22	10.1

Next, description will be given of a case that the specimen cartridge and the specimen cartridge tilting rod are brought into contact with each other on a warped contact surface. Figure 4 is a schematic perspective view of the contact surface 7 with the rod, while Figure 2 is the plan view of the specimen cartridge portion.

Lines A-B and X-Y orthogonally intersect each other on a horizontal plane, and however, points D and E are raised relative to the horizontal plane while points C and F are lowered relative to the horizontal plane. A surface CEFD is a warped surface (of saddle type) as a whole. In case of driving the rod 3 in the vertical direction up or down to cause the tilting specimen cartridge 1 to rotate or tilt about the tilting pivots 4-4 in Fig. 2, the rod 3 is also moved left or right on this warped surface 7 simultaneously. When the specimen cartridge is held flat (Fig. 5(b)), the rod 3 contacts on the line A-B so that, even if the rod length 3 is changed due to thermal expansion, the angle of inclination or tilting of the cartridge 1 is not changed because the direction of the rod is parallel to the contact surface (A-B in (b) of Figure 5). The (a) and (c) of Figures 5 illustrate the cases that the rod 3 is moved up and left or down and right. In case that the rod 3 is moved up and left (as shown in (a) of Figure 5) the rod 3 contacts on the line C'-D', while in case that the rod is moved down and right (as shown in (c) of Figure 5) the rod contacts on the line E'-F'. (The line C'-D' is between A-B and C-D, and the line E'-F' is between A-B and E-F.)

If the contact surface were flat in all area, change in length of the rod 3 caused by thermal expansion would result in change of the angle of or tilting except when the specimen cartridge 1 is flat ((b) of Fig. 5). According to the present invention, however, since the contact surface is the warped surface, the contact line C'-D' or E'-F' is always kept parallel to the rod 3. Therefore, even if the rod length changed due to thermal expansion, the tilting angle can be kept unchanged for all tilting angles.

In this way, by moving the rod 3 up and left or down and right, it is possible to tilt the specimen cartridge 1 upwards or downwards accurately within  $\pm 15^\circ$ . (The lines C-D and E-F are inclined downwards and upwards by  $\pm 15^\circ$  to the line A-B).

Next, description will be given of a power supply line for a specimen cartridge heater.

As shown in Figure 2, a lead wire 50 is extended to the tip end portion of the rod 3 (instead, the rod portion 3 may be made of electrically conductive material while coating an outer surface thereof with insulating material except the tip end portion which is brought into contact with the specimen cartridge 1 for passing current to the specimen cartridge) and another lead wire 51 is extended to one end of the spring 6 (instead it may be extended not to the end portion of the spring 6 but to a spring support member for passing current) so that power is supplied to a heater provided in the specimen cartridge 1.

Method of wiring the leads is not limited to the described one and it is also possible to connect the leads directly to the rod 3 and the spring from the outside of the frame 5.

The applied voltage for the heater is 2.0 mV and the current is 1.15 A. Temperature control is performed by means of the voltage-time control method. Since the specimen cartridge 1 is kept in contact with the frame 5 only by means of the rod 3, spring 6 and sapphire pivots 4, heat conduction therebetween can be minimized.

Figure 6 is a sectional view of the specimen cartridge according to an embodiment, in which the specimen cartridge has a stacked structure including a metal plate 15 and a ceramic heater 16. The structure of Figure 6 makes it possible to provide a uniform temperature distribution. As the ceramic heater 16, a plate heater was used.

Figure 7 shows an overall construction of a transmission electron microscope (TEM) in which a double tilt heating specimen holder of the present invention is incorporated. As shown in Figure 7, above a sample or specimen holder unit 22 are arranged an electron gun 18 serving to produce an electron beam, a gun valve 19, a condensor lens unit 21 and a condensor less aperture means 20, while below the specimen holder unit 22 are arranged an objective less unit 31, a selected area aperture means 30, an image forming lens unit 23, a viewing chamber 29, a camera chamber 27 and the like. Besides, it is possible to observe the state of specimen by a magnifier 25 and a monitoring device 33, to adjust the specimen observing position by operating a specimen moving knob 24, and to adjust the specimen position or to take a picture by control panels 26, 28 provided right and left or a controller 32.

With this TEM, observation of a specimen was made actually by making use of the double tilt specimen holder according to an embodiment of the present invention. The specimen used was made of polycrystalline silicon which was thinned by means of an ion milling. After the specimen was inserted in the electron microscope, supply of the current to the specimen cartridge heater was increased gradually until the specimen was heated up to 200 °C. Acceleration voltage of electrons was 200 kV and observation was made with a magnification ratio of 100,000 to 1,000,000. By rotating the screw rod 12 shown in Figure 1, the fine movement shaft 10 was moved to decide the observing position in one direction (X axis) on the horizontal plane. The observing position in the other direction (Y axis) on the horizontal plane was decided in the same manner as that in the X-axis direction by rotating another screw rod (not shown) provided in the vicinity of the specimen holder of Figure 1. In case of the polycrystalline silicon, lattice image thereof appear clearly at various angles of inclination or tilting. Under the conditions that the specimen was heated to 200 °C and the electron beam was converged to have a diameter of 2 nm, characteristic X-rays from various zones were analyzed. As a result, it was observed that phosphorus (P), atoms of impurity, segregated at grain boundaries.

Further, referring to Figure 1, as the specimen holder 8 is inserted in the column of the electron microscope, it abuts against the connecting or bearing rod 9 of the fine adjustment device and comes to a halt. The fine adjustment device comprises the fine movement shaft 10, the lever the such as bell crank 11, the screw rod 12, the connecting rod 9 and the like. An example of the specimen holder 8 was made of phosphor bronze. The phosphor bronze has a large thermal conductivity (62.9 W/mK) so that the temperature distribution of the specimen becomes uniform.

On the other hand, the connecting rod 9 is made of SUS304 the thermal conductivity of which is small (15.1 W/mK).

As a result, according to the example the present invention, even in case of heating the specimen up to 200 °C, the drift of the specimen was reduced to less than  $\pm 0.1$  nmsec three hours after in contrast to the conventional arts. Summarizing the results described above, Table 2 shows the embodiments of the invention in comparison with the conventional arts.



Table 2

			Connecting Rod	Specimen Holder
	Conventional Art (1)	Material	Cordierite	
		Thermal Conductivity (W/mK)	1.7	
	Conventional Art (2)	Material	Phosphor Bronze	
		Thermal Conductivity (W/mK)	60	
	Example 3	Material	SUS304	Phosphor Bronze
		Thermal Conductivity (W/mK)	15.1	60
	Example 4	Material	Sialon	Titanium
		Thermal Conductivity (W/mK)	10.1	22

Table 2 shows the thermal conductivities of the materials used in the conventional arts and the examples of the invention, while Figure 8 shows the changes of the amount of drift with the lapse of time according to the conventional arts and the example of the present invention. In Figure 8, a straight line (a) represents the conventional art (1) in which since the specimen holder is made of poor thermal conductor the specimen holder does not reach the thermal equilibrium within observation. In Figure 8, a curve (b) represents the conventional art (2) in which since the holder is made of highly thermally conductive material the thermal equilibrium can be reached in a short time, and however, since the connection rod is also made of highly thermally conductive material, the temperature becomes high to increase the drift. In Figure 8, a curve (c) represents the example of the invention in which, since the specimen holder is made of highly thermally conductive material and the connecting rod is made material of much lower thermal conductivity the thermal equilibrium can be reached in a short time and the drift is small after reaching the equilibrium.

As a result of the experiment using the examples of the present invention, the specimen drift became  $\pm 0.1$  nm/sec an hour and a half after.

Now, the power supply line for the tilting specimen cartridge heater according to embodiments of the present invention will be described by referring to Figures 9. A first structure is that the power is supplied

through a lead portion 98 coming in contact due to elastic force and a tilting pivot 96. The electric current flows in from the lead portion 98 to the heater of a specimen support 91 such as a specimen cartridge and then flows out to the outside through the tilting pivot 96 (Figure 9A). A second structure is that the power is supplied through a rod 93 and a spring 95. The electric current flows in from the rod 93 to the heater of the specimen cartridge 91 and then flows out to the outside through the spring 95 (Figure 9B). The applied voltage for the heater is 2.0 V and the current is 0.25A. Temperature control is performed in accordance with the current-time control method. In such structure, it is possible to measure the temperature of the specimen cartridge by attaching a thermocouple 99 to the tip end of the conductive spring as shown in Figure 10. The specimen cartridge can be formed to have a stacked structure including a metal plate and a thin film heater, and a uniform temperature distribution can be actualized.

Further, in the structure of Figure 9A, the specimen cartridge is of a triple-layer structure including metal, ceramic heater and metal.

In Figures 9A, 9B and 10, reference numeral 92 denotes a specimen, 94 denotes a pivot and 97 denotes an outer frame.

Lastly, description will be given results of actual observation and measurement using the electron microscope according to the embodiment of the invention. The specimen of SUS316L which is a material for nuclear facilities was thinned by means of a TEM pole. After the specimen was inserted in the electron microscope, supply of the current to the specimen cartridge heater was increased gradually until the specimen was heated up to 200°C. Acceleration voltage of electrons was 200 kV and observation was made with a magnification ratio of 100,000 to 1,000,000. By rotating the screw rod, the fine movement shaft was moved to decide the observing position in one direction (X axis) on the horizontal plane. The observing position in the other direction (Y axis) on the horizontal plane was decided in the same manner by rotating another screw rod provided in the vicinity of the specimen holder. In this way, the lattice image was photographed.

As other embodiment, there was prepared a specimen holder of coupling structure in which the high temperature part of the specimen holder is made of heat insulating material and the low temperature part thereof is made of low thermal expansion material. This was useful in suppressing the rise of the temperature of the specimen holder to be lower than the specimen temperature.

According to the present invention, particularly in the heating specimen holder for the electron microscope, it becomes possible to control the specimen position and the tilting angle with high accuracy, and the amount of drift of the specimen position can be controlled less than 0.1 nm/sec., thereby enabling highly accurate observation and measurement with the electron microscope.

#### Claims

1. An electron microscope specimen holder having a specimen support (1) within an outer frame (5), wherein the thermal conductivity of the material of said specimen support is greater than that of material of said outer frame.
2. The specimen holder of claim 1, wherein the coefficient of thermal expansion of the material of said outer frame (5) is in the range of  $5.0 \times 10^{-7} \sim 1.2 \times 10^{-5}/K$ .
3. An electron microscope specimen holder according to claim 1, wherein said specimen support (1) is made of a material selected from a group essentially consisting of titanium and stainless steel and said outer frame (5) is made of a material selected from a group essentially consisting of sialon, cordierite and titanium.
4. An electron microscope specimen holder comprising:
  - an outer frame (5),
  - a specimen support (1) within said frame, and
  - tilting pivots (4) serving to incline said specimen support relative to said frame,
  - wherein the thermal conductivity of the material of said specimen support (1) is greater than that of the material of said frame (5) and the coefficient of thermal expansion of the material for said tilting pivots (4) is in the range of  $5.0 \times 10^{-7} \sim 1.2 \times 10^{-5}/K$ .
5. An electron microscope specimen holder in which a specimen set on a specimen support (1) is moved in a plane and driven rotatively, wherein a rod (3) serving to control an angle of said specimen support is in contact with said specimen support on a warped surface (7).

6. An electron microscope specimen holder comprising:  
a specimen support (1),  
a rod (3) serving to control an angle of said specimen support, and  
a pressure spring (6),  
5 wherein said specimen support (1) is of a double-layer structure including a platform on which a specimen is to be placed and a heater, and said rod (3) and said pressure spring (6) serve as a power supply line for said heater.
7. The specimen holder of claim 1, wherein said specimen support (1) has a stacked structure including a  
10 metal portion and a ceramic heater.
8. An electron microscope specimen holder which has a coupling structure including a high temperature part and a low temperature part, wherein said high temperature part is made of heat insulating material and said low temperature part is made of low thermal expansion material.
- 15 9. An electron microscope specimen holder having a specimen support, wherein a thermal conductivity of material of an inner part of said specimen support is greater than that of material of an outer part thereof.
- 20 10. An electron microscope specimen holder in which specimen support is disposed within an outer frame and is to be heated in use, wherein a temperature gradient between said specimen support and said frame is steep when said specimen holder is in use.
- 25 11. An electron microscope specimen holder in which a position of a specimen is changed, wherein an amount of change of said specimen position is in the range of 0.02 ~ 0.10 nm/sec.
12. An electron microscope in which the electron microscope specimen holder according to Claim 11 is mounted.
- 30 13. An electron microscope specimen holder used for observing a state of a specimen placed on a specimen support by applying an electron beam to said specimen, said specimen holder comprising:  
a heater disposed at a lower part of said specimen support for serving to heat the specimen on said specimen support up to a predetermined temperature;  
a rod for controlling an angle of said specimen support; and  
35 a pressure spring for supporting said specimen support,  
wherein said rod has a shape that makes a contact surface between said rod and said specimen support vertically stationary, and power is supplied to said heater through said rod and said pressure spring.
- 40 14. A transmission electron microscope comprising:  
an electron gun serving to produce an electron beam;  
an optical system serving to apply a predetermined amount of said electron beam over a predetermined area;  
a specimen holder on which a specimen is to be placed so as to be observed as being applied  
45 with said electron beam; and  
another optical system and a control system for observing a state of the specimen on said specimen holder,  
wherein said specimen holder has a structure that a specimen support is disposed within an outer frame, a thermal conductivity of material of said specimen support is greater than that of material of  
50 said frame, and said specimen support is provided at a lower part thereof with a heater to which power is supplied through a pressure spring serving to hold said specimen support and through a rod serving to adjust a tilting of said specimen support.
- 55 15. An electron microscope heating specimen holder having a specimen holder and a connection rod, wherein a thermal conductivity of material of said connecting rod is smaller than that of material of a main body of said specimen holder.

EP 0 538 861 A1

16. An electron microscope specimen holder according to Claim 15, wherein the thermal conductivity of said bearing rod is in the range of 15 ~ 20 W/mK.
- 5 17. An electron microscope specimen holder having a specimen holder for supporting a specimen and a connecting rod serving to finely control a position of said specimen, wherein said specimen holder is made of phosphor bronze and said connecting rod is made of SUS304.
- 10 18. An electron microscope specimen holder in which a heater for heating a specimen is disposed at a lower part of a specimen support serving to support said specimen, wherein said heater is a plate heater made by forming a film of high resistance metal on a plate-like insulating member by means of vapor deposition or sintering.
- 15 19. An electron microscope specimen holder having a heater formed on an insulating member and serving to heat a specimen, wherein said heater is a plate heater made by filling a groove formed in a plate-like insulating member with high resistance metal or by sandwiching high resistance metal between plate-like insulating members.
- 20 20. An electron microscope specimen holder having a structure that a thermocouple is in contact with a specimen support, wherein said thermocouple is disposed at a tip end of an elastic lead portion.
- 25 21. An electron microscope in which the electron microscope specimen holder according to Claim 15 is mounted.
- 30
- 35
- 40
- 45
- 50
- 55

FIG. 1

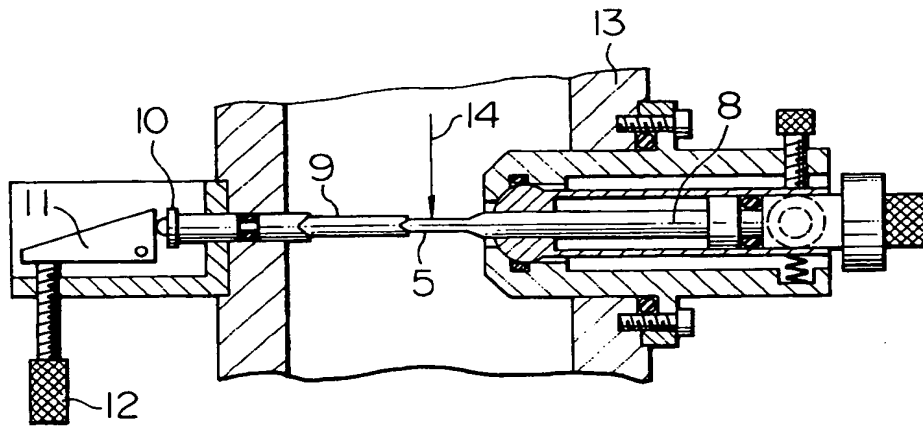


FIG. 2

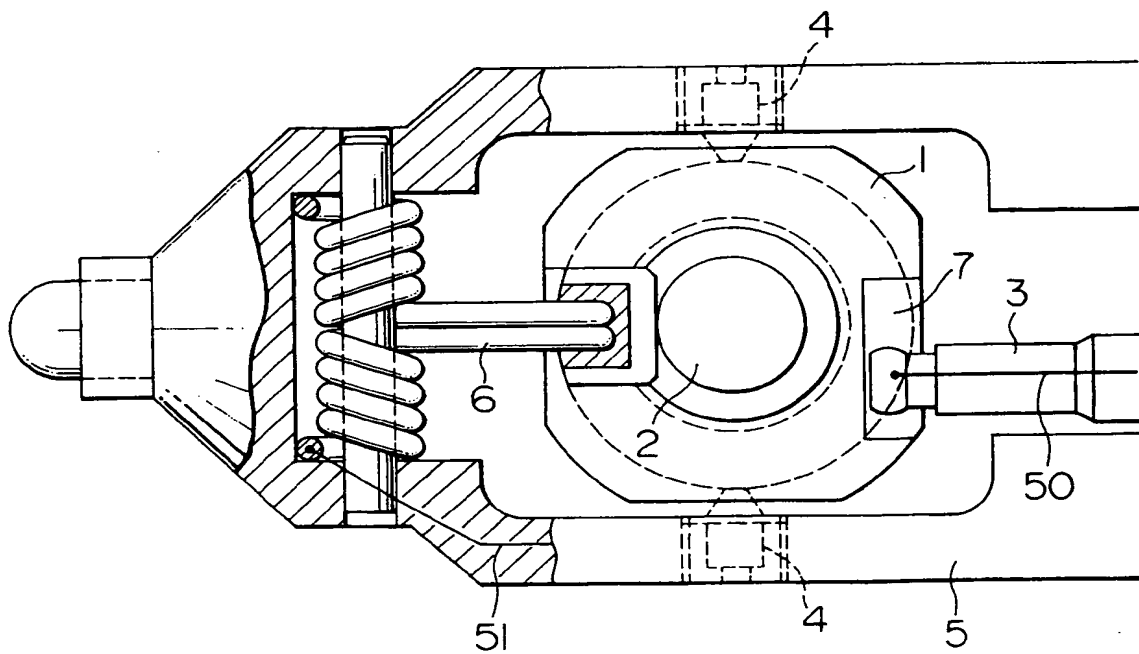


FIG. 3A

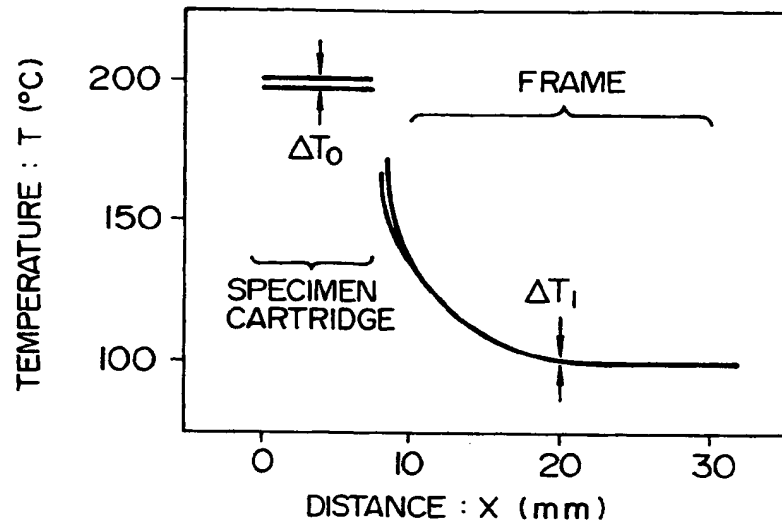


FIG. 3B

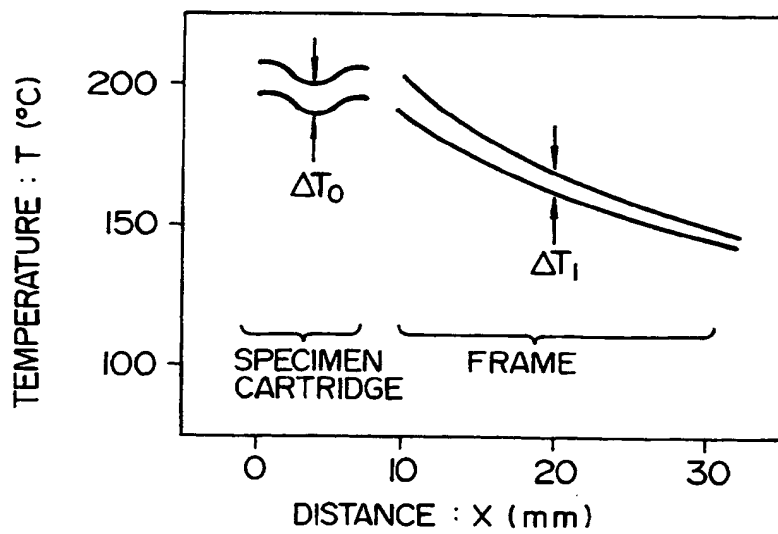


FIG. 4

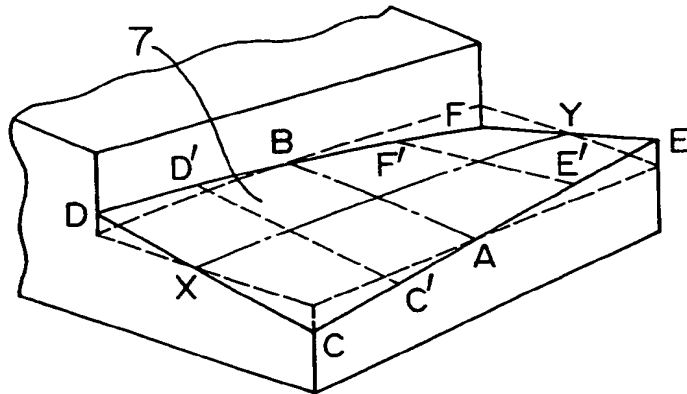


FIG. 5

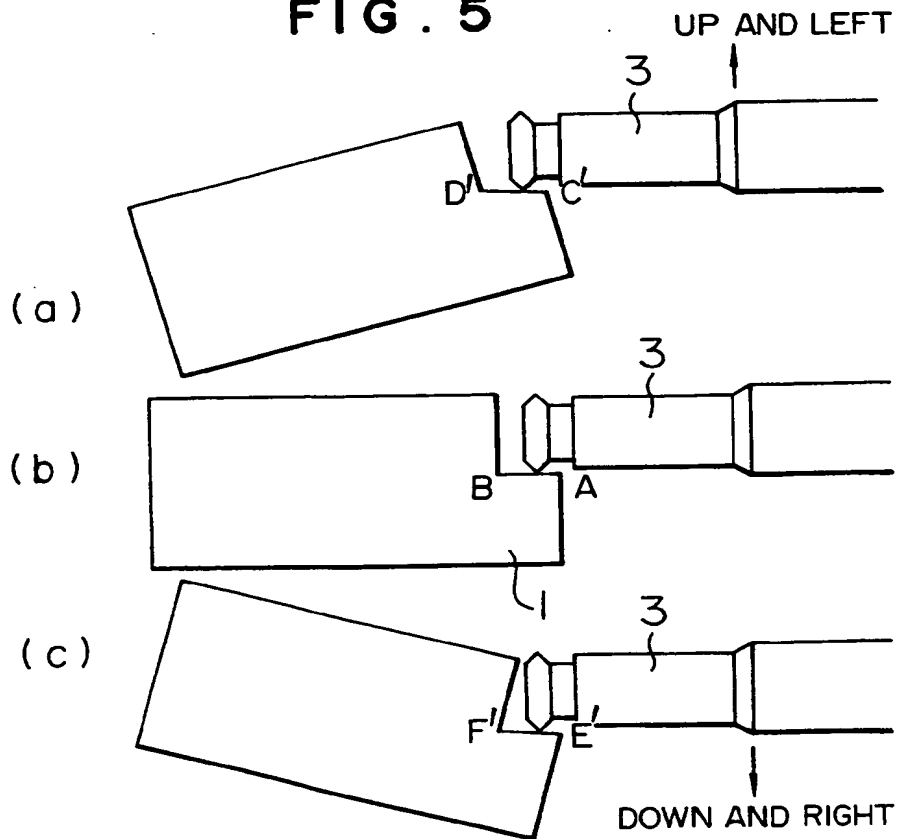


FIG. 6

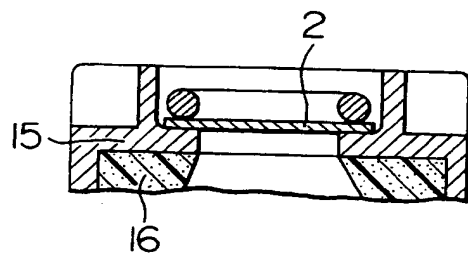


FIG. 7

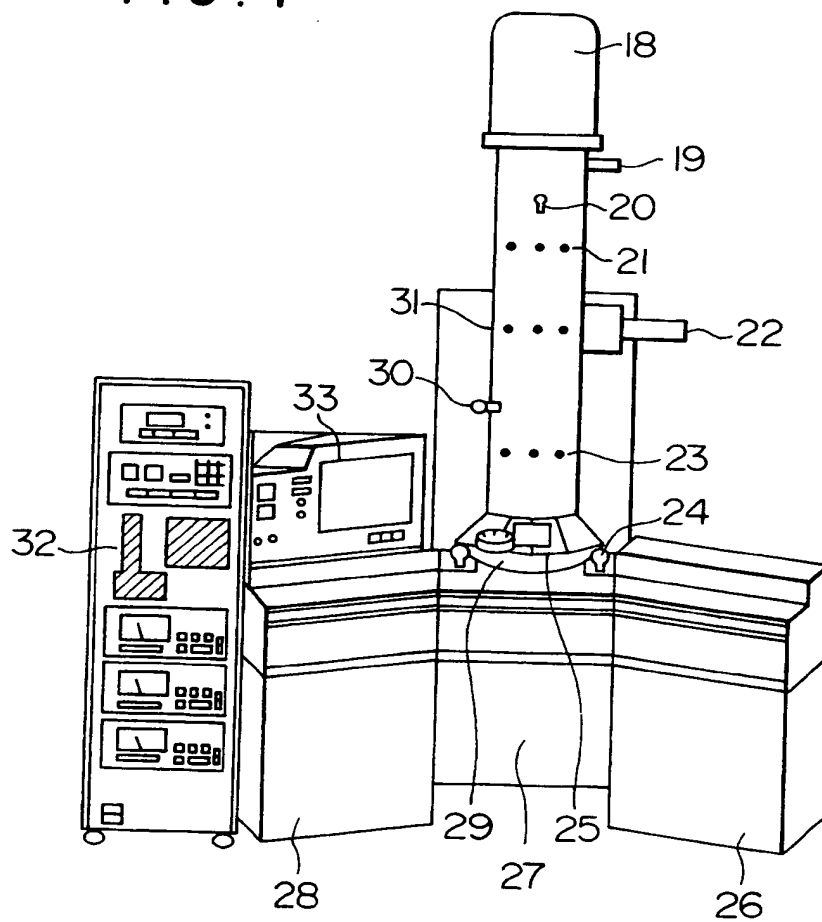




FIG. 8

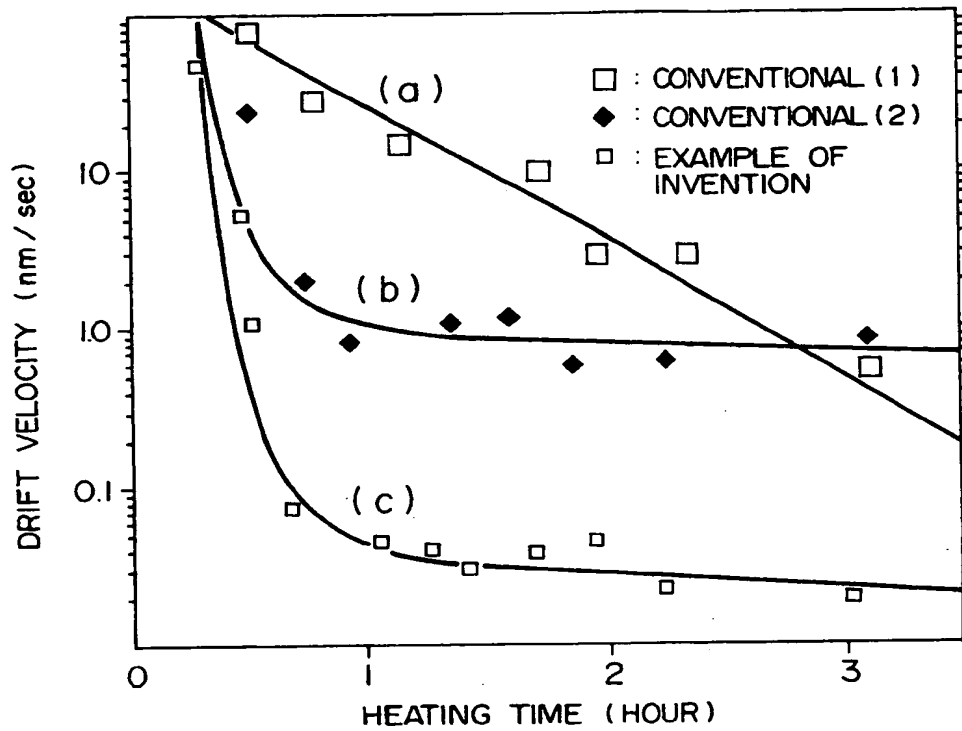


FIG. 10

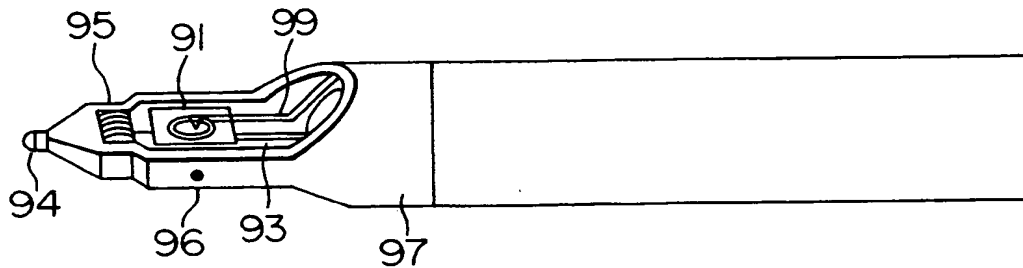


FIG. 9A

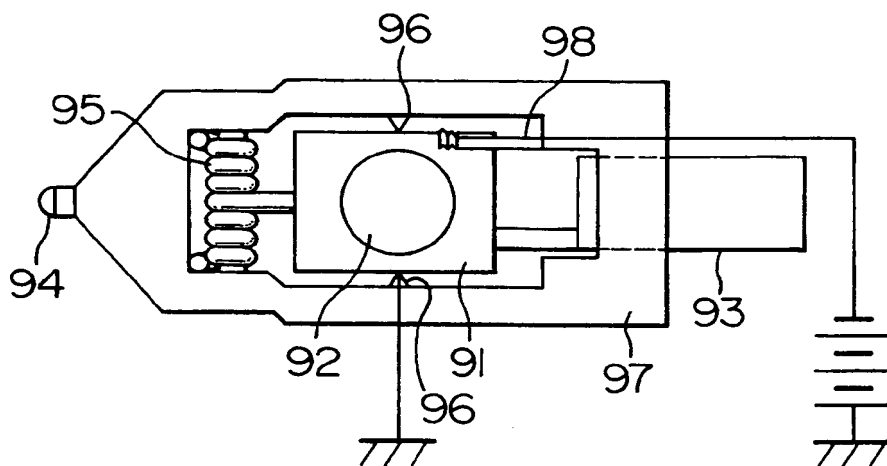
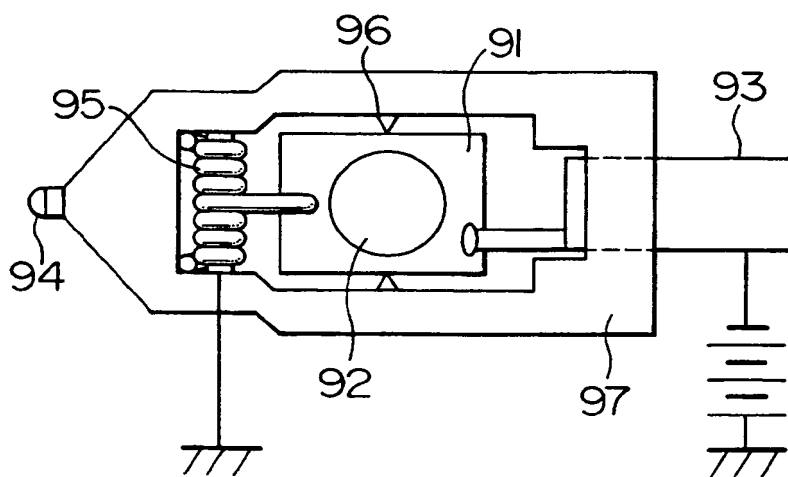


FIG. 9B





European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number

EP 92 11 8098

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	US-A-4 996 433 (JONES ET AL.) * column 3, line 1 - column 4, line 23; figures * ---	1,4,9, 14,17	H01J37/20
A	EP-A-0 427 326 (N.V. PHILIPS'GLOEILAMPENFABRIEKEN) * column 2, line 37 - line 50 * * column 3, line 26 - column 4, line 44; figures * ---	1,6,14	
D,A	US-A-4 703 181 (SWANN ET AL.) * abstract; figures * * column 5, line 4 - line 23 * ---	1	
A	JOURNAL OF PHYSICS E. SCIENTIFIC INSTRUMENTS vol. L5, no. 2, February 1982, BRISTOL GB pages 184 - 186 J.A. EADES 'A helium-cooled specimen stage for electron microscopy.' * the whole document * ---	1	
A	DE-A-2 126 625 (SIEMENS A.G.) * the whole document * -----	5,13,15, 16,21	H01J
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 26 JANUARY 1993	Examiner SCHAUB G.G.
<b>CATEGORY OF CITED DOCUMENTS</b> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document			

**THIS PAGE BLANK (USPTO)**